

Final Report

3M Brookings Ethylene Oxide Abator Engineering Test

LIMS Project Number: E16-0878
Method or Regulatory Requirement: ETS-8-031

Testing Laboratory

3M Environment, Health, Safety and Sustainability
Environmental Laboratory

3M Environmental Laboratory

Levon Trainor
3M Center
260-05-N-17
1-651-736-5065
ltrainor@mmm.com

3M Brookings

Paul Peterson
601 22nd Ave South
Brookings, SD 57006
1-605-696-1445
p-peterson@mmm.com

The laboratory's quality system has been audited and was found to be in conformance with the EPA GLPs (40 CFR 792) as well as ANSI/ISO/IEC 17025:2005 by an independent assessment. The specific test included in this report is not on the lab's scope of accreditation.

Table of Contents

1 Introduction / Summary3

2 Methods- Analytical and Preparatory4

3 Analysis5

4 Data/Sample Retention5

5 Conclusion5

6 Signatures6

7 Attachments7

3M EHSS Environmental Laboratory – Destruction Efficiency Engineering Test

Report Author: Levon Trainor

Analytical Team: Levon Trainor, Kelly Sater

3M Brookings Ethylene Oxide Abator Engineering Test**LIMS Project Number:** E16-0878**Date of Report:** Date of Last Signature**1 Introduction / Summary**

The destruction efficiency of the Ethylene Oxide abator at 3M Brookings was evaluated by Extractive FTIR Spectroscopy. The inlet and outlet gases of the abator were sampled simultaneously with two MKS FTIR spectrometers measuring the gas phase concentrations of Ethylene Oxide. Sampling was performed for the duration of 3 runs. Destruction efficiency was determined using inlet & outlet concentrations - averaged over the portion of the run when ethylene oxide was above the detection limit of 0.24PPMv. Inlet and Outlet airflows were assumed to be equal.

Formaldehyde was also observed in the outlet stream with a maximum concentration of 3.1 PPMv.;

1.1 Destruction Efficiency Results and Discussion**Table 1 Average Concentration and Destruction Efficiency Results**

	Run 1	Run 2	Run 3
Date	11/8/2016	11/8/2016	11/8/2016
Start Time	8:35	11:00	12:45
Conditions	Standard	Standard	Standard
Inlet Concentrations (ppmV)			
Ethylene Oxide	820	1000	1100
Outlet Concentrations (ppmV)			
Ethylene Oxide	1.9	1.2	0.74
Ethylene Oxide Destruction Efficiency	99.8%	99.9%	99.9%

2 Methods- Analytical and Preparatory

Analysis was performed according to a procedure of ETS-8-31.3 “Measurement of Vapor Phase Compounds by Fourier Transform Infrared (FTIR) Spectrometry”, which is based on NIOSH 3800 and EPA Method 320.

The project quality level for this study was designated as “Level Two: Quantitative Monitoring”. Project Quality Level 2 (PQL 2) is appropriate for emission factor estimates and non-compliance test measurements. PQL 2 is appropriate when the project objectives specify the data will not be incorporated in compliance tests of manufacturing emissions, but can be used in certain environmental permitting and regulatory activities such as emission factor estimation.

2.1 Instrumentation

An FTIR with a 5.11 meter nominal pathlength gas cell was used for the analysis. Table 2 gives sampling and configuration parameters of the instrument(s) used:

Table 2 Instrument Parameters

Instrument Name	4MKS	6MKS
Model	MKS MG2030	MKS MG2030
Date Analyzed	11/8/2016	11/8/2016
Nominal Pathlength (m)	5.11	5.11
FTIR Cell Temperature (°C)	35	35
Number of Co-added Background Scans	128	128
Number of Co-added Sample Scans	64	64
Scan Range (cm-1)	650–4500	650-4500
Resolution (cm-1)	0.5	0.5

2.2 Calculations

2.2.1 AutoQuant Results

Results generated using the AutoQuant™ (v4.5) or MG2000 (v7.2) software are reported in ppmv (parts per million by volume). The software was used in conjunction with Midac, EPA, PNNL, MKS, and 3M library reference spectra, and manual subtraction of reference spectra in Thermo GRAMS/AI and/or MG2000.

These results are converted to µg using the following equation:

$$\mu g = \frac{\text{Concentration (ppm}_v\text{)} \times \text{Sample Gas Volume (L)} \times \text{Pressure (atm)} \times \text{Molecular Weight } \left(\frac{g}{mol}\right)}{0.08206(L \times atm \times K^{-1} \times mol^{-1}) \times \text{Cell Temperature (K)}}$$

Where Sample Gas Volume (L) = total chamber compressed house air purge gas volume during sample off-gassing or the volume of the gas cell.

2.2.2 Manual Subtraction

The concentration of a target analyte in a sample FTIR spectrum was verified using manual subtraction of a reference spectrum from the sample spectrum by means of Thermo GRAMS or MG2000 software. The relative fraction of the reference spectrum, or subtraction factor, is then used to calculate the concentration of the sample in ppmv using the following equation.

$$ppm_v = \frac{\text{subtraction factor} \times \text{reference concentration at cell temp (ppm}_v \cdot m\text{)}}{\text{pathlength of cell (m)}}$$

2.2.3 Limit of Quantitation

The limit of quantitation was estimated by manual addition of the analyte quantitative reference spectrum to the sample spectrum. Using the Thermo GRAMS or MG2000 software program, the reference spectrum was added until the analyte signal was approximately two times greater than the surrounding noise. The resulting addition (negative subtraction) factor was used to calculate a ppmv concentration using the equation listed in 2.2.2.

3 Analysis

3.1 Calibration

The instrument was calibrated using a 20.0 ppm certified (see Attachment 7.4) standard of ethylene (cylinder # SG9182087BAL). The instrument gas cell pathlength was determined before and after sampling. (see Attachment 7.3)

3.2 Blanks

Before and after each sample run, the sample-cell was checked for contaminants.

4 Data/Sample Retention

This report and all associated data will be archived and retained according to record retention policy.

5 Conclusion

Matrix spiking was not required for this project. Therefore the uncertainty of the gas phase concentration of the given chemicals as measured using FTIR is +/- 17% and is based on 2 times the standard deviation of the most recent 54 recovery values measured in the ISO 17025 FTIR proficiency testing of 3M Environmental Lab FTIR operators.

Results are only valid for the run conditions from the day of testing.

6 Signatures

Levon Trainor, Project Engineer

Brian Mader, Environmental Laboratory Management

7 Attachments

7.1 Sample Collection Data Sheet(s)

7.2 AutoQuant/MG2000 Methods

The MG2000 method will be archived with this report.

7.3 Pathlength Determination and Calibration Check

Inlet	Spectrum	Date	Time	ETHYLENE 35C	
Pretest	6MKS_0007.LAB	11/8/2016	7:45:38	19.7962	
	6MKS_0008.LAB	11/8/2016	7:46:38	19.817695	
	6MKS_0009.LAB	11/8/2016	7:47:38	19.74312	
	6MKS_0010.LAB	11/8/2016	7:48:38	19.76862	
	6MKS_0011.LAB	11/8/2016	7:49:38	19.818126	
AVG				19.7887522	
Inlet	6MKS_0376.LAB	11/8/2016	14:01:18	19.976921	
PostTest	6MKS_0377.LAB	11/8/2016	14:02:18	20.01599	
	6MKS_0378.LAB	11/8/2016	14:03:18	19.989868	
	6MKS_0379.LAB	11/8/2016	14:04:18	19.918967	
AVG				19.9754365	
Pre vs Post % Difference				<u>-0.94338591</u>	
	Spectrum	Date	Time	ETHYLENE 35C	
Outlet	4MKS__0013.LAB	11/8/2016	7:40:38	19.600812	
Pretest	4MKS__0014.LAB	11/8/2016	7:40:53	19.542298	
	4MKS__0015.LAB	11/8/2016	7:41:08	19.57544	
	4MKS__0016.LAB	11/8/2016	7:41:23	19.563096	
	4MKS__0017.LAB	11/8/2016	7:41:38	19.588237	
AVG				19.5739766	
Outlet	4MKS__0341.LAB	11/8/2016	13:57:15	19.429483	
PostTest	4MKS__0342.LAB	11/8/2016	13:57:30	19.492449	
	4MKS__0343.LAB	11/8/2016	13:57:45	19.542662	
	4MKS__0344.LAB	11/8/2016	13:58:00	19.505138	
	4MKS__0345.LAB	11/8/2016	13:58:15	19.46146	
AVG				19.4862384	
Pre vs Post % Difference				<u>0.448239015</u>	

7.4 Calibration Gas Certification

Oxygen Service Company, Inc.

E16-0878

"An Employee Owned Company"

1111 PIERCE BUTLER RTE

ST. PAUL, MN 55104

(651)644-7273

FAX(651)644-2973

Certificate of Analysis

11719-33546

PURCHASE ORDER : 14-329TK

CYLINDER # SG9182087BAL

COMPONENT	CAS NUMBER	REQUESTED CONCENTRATION	ACTUAL CONCENTRATION	UOM	ACCURACY +/-
ETHYLENE	74-85-1	Mole 20	Mole 20.0	ppm	1%
NITROGEN	7727-37-9	Balance	Balance	%	

METHOD OF ANALYSIS : GAS CHROMATOGRAPHY/GRAVIMETRIC

CYLINDER PRESSURE : 2015 PSIA

CYLINDER CONTENTS : 138 SCF

SHELF LIFE : 36 MONTHS

PRODUCED : 11/25/2014

EXPIRES : 11/25/2017

THIS MIXTURE WAS MADE TO A MINIMUM OF +/-1% ACCURACY USING SCALES THAT HAVE MONTHLY CALIBRATION CHECKS FOR PROCESS CONTROL PURPOSES. SCALES ARE CALIBRATED TWICE A YEAR BY "ALLOMETRICS" WITH N.I.S.T. TRACEABLE WEIGHT SET 610. NIST TRACEABLE TEST 740801-1. THIS CALIBRATION PROCEDURE IS DEFINED IN MIL. STD 45662.

ANALYST




7.5 Peer Review

E16-0878 Peer Review

Review Analysis. A representative subset of sample spectra was peer reviewed by Tim Gutzkow. Specifically a review of the following spectral file(s) was done:

Spectrum	Date	Time	Formaldehyde 35c	EtOv3	Temp (C)	Pressure (Atm)
6MKS_0101.LAB	11/8/2016	9:22:43	-0.218049	1430.735	35.67534	0.900963
TGG Results				1453		
% Difference				-1.6 %		
Spectrum	Date	Time	Formaldehyde 35c	EtOv3	Temp (C)	Pressure (Atm)
4MKS__0072.LAB	11/8/2016	9:24:44	3.801018	4.885826	35.66132	0.938058
			3.862	4.75		
% Difference			-1.6 %	2.78 %		

Signature and Date: _____



1-3-17

7.6 General Project Outline

3M EHS Laboratory General Project Outline

To: Paul Peterson – 3M Brookings
From: Levon Trainor– 3M EHS Laboratory
CC: Brian Mader – 3M EHS Laboratory
 Kelly Sater – 3M EHS Laboratory
 Tim Gutzkow – 3M EHS Laboratory
Date: 11/3/2016
Subject: 3M Brookings Abator Efficiency Test – General Project Outline

➤ Project Objective:

The objective of this project is to conduct a test of destruction efficiency of the 3M Brookings Ethylene Oxide Abator using Extractive FTIR. The testing will be performed to validate that the new catalyst is performing as expected. GC/PID may also be used for testing at discretion of environmental lab field team.

Project Requested by:

Paul Peterson
 3M Brookings
 Dept. Number: 104180
 1-605-696-1445
p-peterson@mmm.com

Project Coordinated by:

Levon Trainor
 EHS Laboratory Analyst
 1-651-736-5065
ltrainor@mmm.com

➤ Test and Reporting Summary

<u>Test Location</u>	3M Brookings
<u>Process/Run Parameters</u>	Normal abator operating conditions with new catalyst replacement.
<u>Target Analytes</u>	Ethylene Oxide
<u>Test Schedule</u>	Testing week of 11/7/2016
<u>Estimated Report Date</u>	12/14/2016
<u>Report to:</u>	Paul Peterson
<u>Reporting Requirements</u>	Detailed Report with supporting appendices
<u>Report Classification</u>	Confidential

➤ Safety

EHS Laboratory personnel will adhere to the stricter of the EHS Laboratory safety policy or the safety policy of the test location.

➤ Project Cost

Actual project costs are determined on a time and materials basis in accordance with the existing 3M EHS Laboratory contract.

Estimated Cost N/A

****Note: A portion or all of the in-house testing cost may be charged directly to the EHS&S Operations department as a corporate operating expense).***

➤ **Test Methods**

1. Speciated FTIR Analysis – Modified EPA Method 320 (3M EL SOP ETS-08-31)

Assigned Project Quality Level: PQL2

The Environmental Laboratory maintains A2LA accreditation to ISO/IEC 17025 for the specific tests/calibrations as listed in A2LA Certificate #2052-01. The test results for FTIR analysis included in this project are covered by this accreditation.